Prepared for

Carus Corporation and
Carus Chemical Company
1500 Eighth Street
P.O. Box 1500
LaSalle, Illinois 61301-3500

U.S. Environmental Protection Agency
Region 5
77 West Jackson Boulevard
Chicago, Illinois 60604

FINAL
SEDIMENT TOXICITY TESTING WORK PLAN
LITTLE VERMILION RIVER

MATTHIESSEN AND HEGELER ZINC COMPANY SITE
LASALLE, ILLINOIS

Prepared by

Geosyntec consultants

engineers | scientists | innovators

134 North LaSalle Street, Suite 300
Chicago, Illinois 60602

Project Number FC1640

18 August 2011

CONTENTS

Section **Page**

1.0 INTRODUCTION 1
2.0 LABORATORY METHODS 2
2.1 *Hyalella azteca* Toxicity Test 2
2.2 *Chironomus dilutus* Toxicity Test 3
2.3 Chemical Analysis 4
3.0 STATISTICAL EVALUATION 5
4.0 FIELD ACTIVITIES 5
4.1 Mobilization and Documentation 5
4.2 Sampling Locations 7
4.3 Sampling Procedures 9
4.3.1 Equipment 9
4.3.2 Sample Collection 9
4.3.3 Decontamination 11
4.3.4 Field Quality Assurance/Quality Control Samples 11
4.3.5 Sample Nomenclature 13
4.4 Packaging and Shipping of Environmental Samples 14
4.4.1 Equipment 14
4.4.2 Procedures 14
4.4.3 Sample Custody 15
5.0 REPORTING AND SCHEDULE 17
6.0 REFERENCES 18

FIGURES

1 Site Location
2 Site and Reference Reach Locations

TABLES

1 Draft BERA Assessment and Measurement Endpoints
2 Summary of Methods for the *Hyalella azteca* Toxicity Test
3 Summary of Methods for the *Chironomus dilutus* Toxicity Test

ACRONYMS AND ABBREVIATIONS

ADD	Average Daily Dose
AFDW	Ash Free Dry Weight
ANOVA	Analysis of Variance
BA	Biological Assessment
BERA	Baseline Ecological Risk Assessment
˚C	Degrees Centigrade
Carus	Carus Corporation and Carus Chemical Company
CFR	Code of Federal Regulations
cm	Centimeter
DO	Dissolved oxygen
DOT	Department of Transportation
ESI	EnviroSystems Inc.
fIBI	Fish community index of biological integrity
FS	Feasibility Study
FSP	Field Sampling Plan
Geosyntec	Geosyntec Consultants, Inc.
HASP	Health and Safety Plan
HAZWOPER	Hazardous Waste Operations and Emergency Response
HHRA	Human Health Risk Assessment
IEPA	Illinois Environmental Protection Agency
L	Liter
LVR	Little Vermilion River
mg	Milligram
mIBI	Macroinvertebrate community index of biological integrity
mL	Milliliter
mm	Millimeter
MS	Matrix Spike
MSD	Matrix Spike Duplicate
ORP	Oxygen Reduction Potential
OU1	Operable Unit 1
OU2	Operable Unit 2
PCB	Polychlorinated Biphenyl
PPE	Personal Protective Equipment
QA	Quality Assurance
QC	Quality Control
RA	Risk Assessment
RI	Remedial Investigation
SHSO	Site Health and Safety Officer
SOP	Standard Operating Procedure
SVOC	Semivolatile Organic Compound
TRV	Toxicity Reference Value
SLERA	Screening Level Ecological Risk Assessment
USEPA	United States Environmental Protection Agency
VOC	Volatile Organic Compound
WA	Work assignment
WP	Work Plan

1.0 INTRODUCTION

Geosyntec Consultants, Inc. (Geosyntec), on behalf of Carus Corporation and Carus Chemical Company (Carus) has prepared this Work Plan (WP) for additional ecological investigation of the Little Vermilion River (LVR) of the Matthiessen and Hegeler Zinc Company Site (Site), which is located in LaSalle, LaSalle County, Illinois (**Figure 1**). In May 2010, a Draft Remedial Investigation (RI) Report was submitted to the United States Environmental Protection Agency (USEPA). A Draft Risk Assessment (RA) was included as an appendix to the RI; the Draft RA presented the human health risk assessment (HHRA), screening level ecological risk assessment (SLERA), and baseline ecological risk assessment (BERA) for the Site. The sediment toxicity testing proposed herein will be conducted in support of the BERA for the LVR.

The Draft BERA evaluated the assessment endpoints and measurement endpoints summarized in Table 1.

Table 1: Draft BERA Assessment and Measurement Endpoints

Assessment Endpoint	Measurement Endpoint
Function and viability of the benthic communities in the LVR	Community assessment and calculation of a macroinvertebrate community index of biotic integrity (mIBI)
Function and viability of the aquatic (fish) communities in the LVR	Community assessment and calculation of a fish community index of biotic integrity (fIBI)
Survival and reproduction of mammalian populations that feed/forage in the LVR	Food chain modeling with Site sediment, surface water, and tissue data to calculate an average daily dose (ADD), and comparison of the ADD to low and high toxicity reference values (TRVs)
Survival and reproduction of avian populations that feed/forage in the LVR	

In a letter dated 25 February 2011, USEPA expressed their position that "…further benthic toxicity testing will be required in order to make a determination on Geosyntec's findings that the macroinvertebrate community of the LVR adjacent to the Site is not significantly different from "background" species diversity measured at the same-stream reach reference."

This WP provides a description of the sediment toxicity testing to be conducted in the LVR. The primary objective of the sediment toxicity testing is to determine whether potential risks, if any, to benthic invertebrate communities in the LVR adjacent to the Site are statistically different from potential risks to benthic invertebrates in an unimpacted (upstream) portion of the LVR. Sediment toxicity tests will be conducted using the following test methods, which are described in further detail in Section 2.0:

- 28-Day *Hyalella azteca* toxicity test for growth and survival; and
- 10-day *Chironomus dilutus* toxicity test for growth and survival.

Potential risks to the benthic invertebrate community of the LVR will be assessed in the Final BERA using a weight-of-evidence approach that integrates the results of the sediment toxicity tests with whole sediment chemistry data and the results of the macroinvertebrate community assessment as reported in the Biological Assessment of the Little Vermilion River Adjacent to the Matthiessen and Hegeler Zinc Company Site (BA) (final revision to be submitted 16 May 2011).

The remainder of this document is organized as follows:

- Section 2: Laboratory Methods
- Section 3: Statistical Evaluation
- Section 4: Field Activities
- Section 5: Reporting and Schedule
- Section 6: References

2.0 LABORATORY METHODS

This section provides a summary of the testing methods used for the samples collected for sediment toxicity testing. As described in detail in Section 4.2, sediment samples will be collected from the three reaches of the LVR adjacent to the Site and one upstream reference reach and submitted to EnviroSystems, Inc. (ESI) laboratories in Hampton, New Hampshire for 28-Day *Hyalella azteca* toxicity tests for growth and survival and 10-day *Chironomus dilutus* toxicity test for growth and survival. Details for each method are described in the following subsections. In addition, control negative tests will be conducted for both test organisms using formulated sediment (USEPA, 2000) and the testing protocols detailed below.

2.1 *Hyalella azteca* Toxicity Test

H. azteca is a burrowing amphipod that is sensitive to contaminants associated with sediment and in direct contact with the sediment (primarily surficial sediment). Based on these characteristics, *H. azteca* was selected for toxicity testing of LVR sediments.

The protocol used for *H. azteca* will be a 28-day day toxicity test for growth and survival, which is generally based on Method 100.4 in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates* (USEPA, 2000). Method 100.4 is a 42-day test that measures survival, growth, and reproduction; thus, the primary deviations from the protocol will be the shortened duration and the exclusion of reproduction as a measurement endpoint. The 28-day study duration was selected based on discussions with USEPA on the appropriate test

duration to ensure capturing potential impacts from sediments and is supported by the scientific literature (Besser *et al.*, 2008). Table 2 below (adopted from USEPA, 2000) summarizes the toxicity test methods.

Table 2 Summary of Methods for the *Hyalella azteca* Toxicity Test

Parameter	Condition or Regimen
Test type	whole-sediment toxicity test with renewal of overlying water
Test duration	28 days
Endpoints measured	survival and growth
Test temperature	23 ± 1 degrees centigrade (˚C)
Illuminance	About 100 to 1000 lux
Photoperiod	16 hours of light to 8 hours of dark
Test chamber	300 (milliliter) mL high-form lipless beaker
Test sediment volume	100 mL
Overlying water volume	175 mL
Renewal of overlying water	2 volume additions per day
Overlying water:	reconstituted water/surface water blend (approximately 50/50)
Overlying water quality	Water quality will be tested for: hardness, alkalinity, conductivity, and ammonia (days 0, 7, 14, 21, and 28); temperature (daily); dissolved oxygen (DO) and pH (days 0, 28, and 3x per week)
Age of organism	7- to 8- days old at the start of the test
Number of organisms/chamber	10
Number of replicates	8
Feeding	1.0 mL of yeast/trout chow/alfalfa suspension daily
Aeration	none, unless DO in overlying water <2.5 milligrams (mg) per liter (L)
Test acceptability	Minimum mean control survival ≥ 80% on day 28 and measurable growth of control test organisms. Note: for the growth endpoint, measurements will be based on the dry weight of surviving adult amphipods.

2.2 *Chironomus dilutus* Toxicity Test

C. dilutus is a burrowing insect that is sensitive to contaminants associated with sediment and in direct contact with the sediment (a few centimeters deep). Based on these characteristics, *C. dilutus* was selected for toxicity testing of LVR sediments.

The protocol used for *C. dilutus* will be a 10-day day toxicity test for growth and survival. The protocol will be based on Method 100.2 in *Methods for Measuring the Toxicity and Bioaccumulation of Sediment-Associated Contaminants with Freshwater Invertebrates* (USEPA, 2000). Table 3 below (adopted from USEPA, 2000) summarizes the toxicity test methods.

Table 3 Summary of Methods for the *Chironomus dilutus* Toxicity Test

Parameter	Condition or Regimen
Test type	whole-sediment toxicity test with renewal of overlying water
Test duration	10 days
Endpoints measured	survival and growth
Test temperature	23 ± 1 ˚C
Illuminance	About 100 to 1000 lux
Photoperiod	16 hours of light to 8 hours of dark
Test chamber	300 mL high-form lipless beaker
Test sediment volume	100 mL
Overlying water volume	175 mL
Renewal of overlying water	2 volume additions per day
Overlying water:	surface water
Overlying water quality	Water quality will be tested for: -hardness, alkalinity, conductivity, pH, and ammonia at days 0 and 10 -temperature and DO daily
Age of organism	Second- to third-instar larvae (about 10-d-old larvae; all organisms third instar or younger with at least 50% of the organisms at third instar)
Number of organisms/chamber	10
Number of replicates	8
Feeding	1.5 mL contains 6-mg TetraMin flake fish food suspension
Aeration	none, unless DO in overlying water <2.5 mg/L
Test acceptability	Minimum mean control survival ≥ 70% on day 10 with minimum mean weight/surviving control organism of 0.48 mg ash free dry weight (AFDW).

2.3 Chemical Analysis

A split sample will be collected in the field from each sediment aliquot and analyzed for potentially Site-related constituents. As detailed below, samples for volatile organic compound (VOC) analysis will be collected independently of other samples. Target analytes for sediment are as follows:

- Total solids, particle size, total organic carbon
- Nitrogen and ammonia
- Site metals (arsenic, cadmium, cobalt, copper, lead, mercury, nickel, silver, zinc)
- Cyanide (total and amenable)
- Organochlorine pesticides
- Polychlorinated biphenyls (PCBs)
- Semivolatile organic compounds (SVOCs)
- VOCs

3.0 STATISTICAL EVALUATION

As indicated in the previous section, the toxicity tests will be considered acceptable if the laboratory control negative samples meet the following criteria:

- For *H. azteca*, minimum mean control survival of 80% or greater on day 28 and measurable growth of control test organisms (i.e., an increase in dry weight of surviving adult amphipods); and

- For *C. dilutus*, minimum mean control survival of 70% or greater on day 10, with a minimum mean weight in surviving control organisms of 0.48 mg AFDW.

Hypothesis testing using analysis of variance (ANOVA) will be used to determine whether statistical difference exists between the toxicity test results for field samples collected from each of three LVR reaches adjacent to the Site as compared to the LVR upstream reference reach. A biologically significant response level of 20% will be used as the threshold for significant effects. Significant ($p<0.05$) control normalized growth and mortality response greater than 20% compared to the reference site may have ecological relevance for the survival of populations in the field (Suter, 1996; and Suter and Tsao, 1996).

Potential risks to the benthic invertebrate community of the LVR will be assessed in the Final BERA using a weight-of-evidence approach that integrates the results of the sediment toxicity tests with previously collected whole sediment chemistry data and the results of the Final BA.

4.0 FIELD ACTIVITIES

4.1 Mobilization and Documentation

Field activities will be scheduled after USEPA review and approval of the WP, but are tentatively scheduled for early summer (June) 2011. Safely accessing the river to collect sediment samples requires wadeable conditions; thus, mobilization is dependent on weather and flow of the LVR. Geosyntec will coordinate with USEPA and their contractor, SulTRAC, to make a "go/no go" decision at least 48 hours prior to field mobilization. Geosyntec will complete the following tasks in preparation for field mobilization:

- develop a field schedule and provide copies to USEPA, SulTRAC, and Illinois Environmental Protection Agency (IEPA);

- contact the receiving laboratory to confirm analytical requirements (e.g., sample size, hold times), obtain sample containers (or schedule appropriate delivery time), and coordinate pick-up of samples;

- obtain field equipment and supplies necessary for sampling; and

- provide copies of relevant project documents (e.g., scope of work, health and safety plan [HASP][1]) to field personnel.

Site access control is important to protect the public from exposure to chemicals at the Site during field activities. All visitors (i.e., field personnel) must check in daily at the Carus facility front office and receive an access badge. The main plant area is fenced and access is controlled by gates. During the work day, access is limited to one open gate (the main entryway); however, this gate is closed at night. All visitors must check in with the Field Manager and Site Health and Safety Officer (SHSO) before being allowed to enter work areas. Visitor information (e.g., affiliation, reason for access, etc.) will be documented in the field log book. Unauthorized visitors will not be allowed to enter work areas. Where applicable, proof of Hazardous Waste Operations and Emergency Response (HAZWOPER) training and evidence of participation in a medical surveillance program will be required before being allowed to enter the work area. Specifically, for on-Site work and off-Site work areas where reference samples for sediment will be collected, visitors will be required to present to the SHSO: (i) a copy of their completion certificates for 40-hour HAZWOPER training and 8-hour refresher training; and (ii) evidence of participation in a medical surveillance program for inclusion in the HASP. All personnel entering the Site will review and sign the HASP and participate in a daily health and safety meeting prior to entering the work areas; the nature of the daily health and safety meeting, with signatures of all participants, will be recorded in the field log book.

The Field Manager will document field activities related to this WP in a single bound field log book with moisture-resistant pages using waterproof ink. The log book will identify the project name, project number, and geographic location of the Site; it will also indicate the name and mobile telephone number of the Field Manager in the event that the log book is lost and recovered. Daily field activities and sampling information will be entered in the log book on serially-numbered pages. At the end of each day's entries, sample collection personnel shall sign and date the entry. Corrections will be made to entries with initialed and dated line-out deletions. A diagonal line will be drawn across the remaining blank space of the last page of each day's entry. All log book lines will be used in sequence, and no blank lines shall remain at the end of the day. All observations will be recorded in sequence. The timeframe covered will be clearly indicated on the front cover and spine by noting the date range of work and investigative phase name.

The log book will be used to document a summary of the day's activities and non-repetitive tasks, including the following:

---

[1] The HASP developed for use during the RI Work Plan (Geosyntec, 2007) will serve as the Site HASP updated to include current project personnel.

- time of arrival and departure from the Site, including lunch breaks;
- names of field team members;
- the nature of the daily health and safety meeting, with signatures of all participants;
- instrument calibration;
- supply deliveries and shipments;
- weather;
- interaction with agency or client personnel;
- incident occurrence and management; and
- any other irregular or ad hoc activities.

The log book will also be used to document water quality measurements (e.g., temperature, conductivity, DO, turbidity, pH, ORP) and to record a description of the sampling locations, including GPS coordinates.

4.2 Sampling Locations

Consistent with the community assessment, sediment samples for toxicity testing will be collected from the three river reaches adjacent to the Site and the upstream reference reach. The four sampling reaches are described below and shown in **Figure 2**.

- *CAR001* – This sample reach is the southernmost (and furthest downstream) reach and is located approximately 0.10 river mile upstream of the 5th Street (State Route 6) Bridge and immediately adjacent to the southern extent of the slag pile located in Operable Unit 1 (OU1).

- *CAR002* – This sample reach is located approximately 0.21 river mile upstream of the 5th Street Bridge and immediately adjacent to the OU1 slag pile.

- *CAR003* – This sample reach is located 0.44 river mile upstream of the 5th Street Bridge and at the northern end of the OU1 slag pile. Though adjacent to the slag pile, CAR003 was established to also measure the potential effects of the City of LaSalle Combine Sewer Overflow (CSO) and abandoned sewer outfall (ASO) discharges associated with Operable Unit 2 (OU2).

- *CAR004 (Reference Reach)* – This sample reach is located approximately 2.32 river miles upstream of the 5th Street Bridge and upstream of the Site.

Three sediment samples will be collected from each Site reach (CAR001, CAR002, CAR003) and from the reference reach (CAR004). Given the significant mobilization efforts to sample within the river and

the potential for changing conditions, sediment sampling locations will be identified in coordination with USEPA and SulTRAC in the field immediately prior to collection activities.

Specific sampling locations are not described herein; however, the following procedure will be used to identify sampling locations.

1. ***Identify Depositional Areas***. Field personnel, wearing personal protective equipment (PPE) specified in the HASP, will wade downstream to upstream within each sampling reach of the LVR sediment probes to identify candidate depositional areas for sampling within each reach. The primary criteria for identifying candidate locations are accessibility (safety, wadeability) and the presence of sufficient sediment to collect a 1 gallon aliquot from the biologically active zone. The biologically active zone is inhabited by infaunal organisms including microbes, meiofauna, and macroinvertebrates and other organisms (e.g., egg and larval stage of fish) that spend all or part of their life cycles associated either within (infaunal) or on (epibenthic) the bottom sediments. The depth of the biologically active zone varies depending on the substrate characteristics present (including particle size fractions, organic matter content, compaction, pore-water geochemistry, and water content) which influence the composition of sediment-associated organisms present. The biologically active zone typically encompasses the top 20 to 40 centimeters (cm) of sediment in freshwater environments (Clarke *et al*., 2001). The majority of benthic organisms will usually be associated with the upper strata (e.g., 15 cm) related to these depth ranges.

2. ***Select Depositional Areas for Sampling***. In coordination with USEPA and SulTRAC, three representative depositional areas within each sampling reach of the LVR will be selected for sediment sampling from the candidate list of depositional areas. To the extent possible, the selected depositional areas will be representative of the length of the reach (e.g., unbiased in terms of stream bank or downstream distance).

3. ***Collect Composite Samples.*** A three- to five-point composite sample will be collected from each depositional area for a total of 9 Site samples and 3 reference samples. Sediment collection procedures are described in the following section.

Selected depositional areas will be photographed, qualitatively described in the field log book, and recorded by field GPS. The number of grab samples required to amass the 1 gallon composite sample will also be recorded in the field log book. The procedure described above will be repeated at each reach immediately prior to sampling, meaning, the depositional areas for sampling will be selected three at a time (per reach) rather than selecting all 12 upon field mobilization. All field activities will be completed at one reach prior to mobilizing to the next reach.

4.3 Sampling Procedures

As described in the previous section, three composite sediment samples will be collected from the biologically active zone of each reach. The composite sample will be amassed from multiple grab samples collected within a defined depositional area. The objective of the sediment sampling is to obtain a representative sample for laboratory analysis of benthic toxicity and for potentially Site-related constituents. The objective requires that the sample be free of unsuitable material and be of sufficient quantity and quality for the selected analytical methods.

The sediment sampling procedure outlined in the following subsections is generally consistent with Standard Operating Procedure (SOP) No. 300 of the RI/FS planning document submitted to USEPA in July 2007 (Geosyntec, 2007).

4.3.1 Equipment

The following equipment is needed for sediment sampling:

- GPS unit
- Water quality meter
- Steel-toed boots, chest-high waders, and other personal protective equipment specified in the HASP
- Decontaminated stainless steel spoons, spatula, Ekman sampler, ponar sampler, and 5-gallon plastic buckets.
- #10 screen (1 millimeters [mm])
- Indelible markers
- Sample containers and VOC sampling syringe (obtained from the laboratory)
- Decontamination supplies (non-phosphate soap, distilled water, tap water, 10% nitric acid, deionized water, methanol, aluminum foil)
- Waterproof field log book and pen

4.3.2 Sample Collection

Once the three depositional areas for sediment sampling have been selected, the samples should be collected in a downstream to upstream fashion within the reach. (As noted above, the depositional areas for sampling within each reach should be identified immediately before sampling (three at a time) and all field activities completed at one reach prior to mobilizing to the next reach.)

The following procedure will be followed for sediment sampling within each depositional area:

1. Don PPE as described in the HASP (note: PPE should be worn at all times while in the river).

2. Select three to five sampling locations within the depositional area from which to collect sediment grab samples.

3. A sediment sample for VOC analysis will be collected at only one sample location within each depositional area. Sediment samples for VOC analysis shall be collected directly from the sampling device using an open barrel, disposable syringe. The syringe will be filled with the lab-specified volumes of undisturbed sediment. The syringe will be shipped to the laboratory for extraction and analysis (i.e., the sediment sample will not be transferred to a separate container in the field). In the event that a field screening technique (e.g., visual staining of sediment or olfactory observation) indicates the presence of VOCs or hydrocarbons, a note will be made in the field log book. Once the VOC sample has been collected, the remaining sample will be processed for other analysis.

4. At each sampling location, collect sediment from the biologically active zone (approximately the top 15 cm of the river sediment unless agreed to by all parties that an alternative depth interval at a specific location is representative – for example in cases where bedrock is encountered at less than 15 cm of sediment depth). Samples will be collected using either a pre-cleaned stainless steel spoon, pole mounted Eckman sampler or a ponar sampler. The exact sampling equipment used will be determined in the field as conditions warrant and in consultation with agency representatives. Samplers may be reused within a given depositional area, as the sediment will be composited and homogenized prior to collecting an aliquot for sampling. However, all equipment must be decontaminated between depositional areas.

5. Remove any vegetation debris (leaves, roots, bark) and large stones/slag from the sample and sieve through a #10 screen (1 mm) such that only the finer sediment material is collected.

6. Allow excess liquids collected along with the sediment sample to decant and place the sieved sample in a plastic bucket.

7. Collect sediment from the remaining sampling locations within the depositional area, and repeat steps 4 through 6 until the necessary amount of sediment has been amassed.

8. Homogenize the composited sediment sample in the plastic bucket.

9. Split samples will be collected from the homogenized sample and placed into the appropriate sample containers. The sediment sample will be analyzed for chemical analysis (except VOCs – see #3) and benthic sediment toxicity (see Section 2.0).

10. Label the sample containers with a sample ID (see Section 4.3.5), date, time, analysis to be performed, and any other pertinent information. Fill out the Chain-of-Custody and store the samples on ice until they are packaged and shipped (see Section 4.4).

11. Detail in the field log book the location of the depositional area, the number of grab samples included in the composite sample, sample ID, time and date, personnel, description of the sample, and other pertinent observations.

At the time of sediment sampling, GPS locations, field measurements of water quality (e.g., temperature, conductivity, DO, turbidity, pH, and oxygen reduction potential [ORP]) will also be measured at each station and recorded in the field log book.

4.3.3 Decontamination

Decontamination of sampling equipment will take place at a centralized area. Samplers may be reused within a given depositional area, as the sediment will be composited and homogenized prior to collecting an aliquot for sampling. However, all equipment must be decontaminated between depositional areas. The following steps, which are consistent with procedures presented in the Phase II Field Sampling Plan (FSP; Geosyntec, 2007), outline the decontamination protocol for sampling equipment:

1. Tap water wash with non-phosphate soap (e.g., Alconox);
2. Tap water rinse;
3. Distilled water rinse;
4. Rinse with 10% nitric acid;
5. Deionized water rinse;
6. Methanol rinse;
7. Deionized water rinse;
8. Air dry; and
9. Aluminum foil wrap.

All disposable and single-use materials and equipment used for decontamination must be disposed of properly. Clothing, tools, buckets, brushes, and all other equipment that is contaminated must be secured in drums or other containers and labeled.

4.3.4 Field Quality Assurance/Quality Control Samples

Quality Assurance/Quality Control (QA/QC) samples are typically collected in the field and submitted to the laboratory along with other environmental samples to evaluate field and laboratory precision and accuracy. Evaluation of QA/QC sample results allows for the quality of the data to be assessed as part of

the overall project QA. The QA/QC samples to be collected at the Site are described below. Trip blank, equipment rinsate blank, filter blank, and field blank samples are used to assess field conditions during sample collection and transport. Duplicates and matrix spike/matrix spike duplicate (MS/MSD) samples are replicate samples used to help assess laboratory precision and accuracy.

- ***Trip Blanks***: Trip blanks are filled with reagent grade water at the laboratory, shipped to the Site with the empty sample containers, and returned to the laboratory with the filled sample containers. Trip blanks are used to determine if VOC samples have been cross-contaminated during shipping and handling.

- ***Temperature Blank***: One temperature blank will also be included with every shipping container from the laboratory to ensure that the samples arrive at acceptable temperatures.

- ***Equipment Rinsate Blanks***: Equipment rinsate blanks will be collected following decontamination of sampling equipment (e.g., buckets, spoons). One equipment rinsate sample will be collected per day. Following decontamination of the equipment, deionized water will be poured over selected sampling equipment and collected for laboratory analysis. The equipment rinsate samples will be analyzed using the same methods used for field samples that day.

- ***Field Blanks***: Field blanks are samples of source water used for decontamination. One field blank sample will be collected for each source of water used for decontamination. Field blanks will be analyzed for metals.

- ***Duplicate Samples***: Duplicate sediment samples are split samples collected in the field. One duplicate sediment sample will be collected and submitted for laboratory analysis. Duplicate samples will be collected after sample sieving and homogenization to evaluate the effectiveness of the homogenization protocol.

- ***MS/MSD Samples***: MS/MSD samples are replicate samples that are spiked with a known concentration of constituents which are then measured as they would be for field samples; the results are used to determine precision and accuracy. One MS/MSD sample will be collected. The volume of sample collected for MS/MSD samples is triple the routine volume: the first aliquot serves as the field sample, the second aliquot as the MS, and the third as the MSD. An exception is for metals; in this case, only double the sample volume is required.

Methods for assigning unique sample names to QA/QC samples are discussed in Section 4.3.5. The unique sample name will be used on the sample containers, sample tags, and Chain-of-Custody Record. Samples will be placed in laboratory-supplied containers and preserved in accordance with the analytical requirements specified by the contracted laboratory.

4.3.5 Sample Nomenclature

The sample identification scheme for field sample collection will be consistent with that used in Phase I and Phase II of the RI and utilize a three-letter project identification code followed by a sample type code, location code, and date details. The general form is as follows:

OU1-aa-bbbbbb-yymmdd

The identification components are described below.

OU1 differentiates samples and locations collected from OU1 from those from OU2.

"aa" indicates the matrix. For sediment samples and associated duplicates, "SE" is the matrix code. For QA/QC samples, the surface water matrix code "SW" will be used in order to have the appearance of a field sample.

"bbbbbb" indicates the sample location and consists of up to six characters. The first three letters of the location code for the sediment toxicity code will be "LVR" (Little Vermilion River) and the last three letters will indicate a specific location. Hyphens will be omitted. Sediment toxicity testing samples will use the location identifications 601 through 612, which will be correlated with the LVR sampling reaches.

"yymmdd" indicates the year, month, and day of the sampling. Single digit months or days will include a leading "0".

QA/QC samples will have a blind naming system to ensure that they are treated the same way as field samples are treated. For all QA/QC samples, use the following fictitious location codes "bbbbbb", each of which indicates a type of QA/QC sample. If multiple QA/QC samples are collected on a given day, append A, B, C, etc. in sequence to the fictitious location name. The fictitious locations are as follow:

- for duplicates, use LVR620;
- for equipment blanks, use LVR621;
- for trip blanks, use LVR622;
- for field blanks, use LVR623; and
- for temperature blanks, use LVR624.

Because the sample ID gives no parent sample information for field duplicates, it is imperative that field documentation record this information so that parent/duplicate data pairings are available after analytical data have been received. For MS/MSDs, use the same sample ID as the parent sample and indicate "MS/MSD" in the comment field on the Chain-of-Custody Record.

4.4 Packaging and Shipping of Environmental Samples

The sediment packing and shipping procedure outlined in the following subsections is consistent with SOP No. 410 of the RI/FS planning document submitted to USEPA in July 2007 (Geosyntec, 2007). The purpose of proper packing and shipping is to protect the integrity of environmental samples shipped for analysis and to ensure that environmental samples arrive at the laboratory in good condition for analysis.

4.4.1 Equipment

The following equipment is needed for sediment packaging and shipping:

- Coolers with return address of the Site office written inside the lid;
- Heavy-duty plastic overbags (cooler size);
- Plastic zip-top bags (small and large);
- Plastic packing tape;
- Duct tape
- Bubble wrap;
- Ice;
- Chain-of-Custody seals;
- Complete Chain-of-Custody Record (see Section 4.4.3); and
- Completed bill of lading or airbill.

The term "Environmental Sample" refers to any sample that has less than reportable quantities of any hazardous constituents according to the Department of Transportation (DOT) 49 *Code of Federal Regulations* (CFR) – Section 172.

4.4.2 Procedures

The follow steps must be followed when packing for shipment:

1. Select a sturdy cooler in good repair. Secure and tape the drain plugs (inside and outside) with duct tape.

2. Be sure the caps on all bottles are tight (will not leak); check to see that labels/tags and Chain-of-Custody Records are properly completed.

3. Double-bag ice in large plastic zip-top bags and properly seal.

4. Place all bottles in separate and appropriately sized plastic zip-top bags and close the bags. Glass bottles should be wrapped in bubble wrap before placing in zip-top bags.

5. Place two layers of large-bubble bubble wrap on the bottom of the cooler.

6. Place a clear, cooler-size overwrap bag in the cooler, place one layer of ice and a temperature blank in the bottom of the bag, and then place the bottles in the bag with sufficient space to allow of the addition of a second layer of ice bags over the sample containers. It is preferable to place glass sample bottles and jars into the cooler vertically. Due to the strength properties of a glass container, there is much less chance for breakage when the container is packed vertically rather than horizontally.

7. Place the second layer of ice bags on top of the samples. Close and securely fasten the top of the large overwrap with tape.

8. Place the complete Chain-of-Custody Record for the laboratory into a plastic zip-top bag, tape the bag to the inner side of the cooler's lid, and then close the cooler.

9. Packing tape shall be wrapped around each end of the cooler two times, and complete Chain-of-Custody seals affixed to the top opposite sides of the cooler half on the tape so that the cooler cannot be opened without breaking the seal. Wrap clear tape over custody seals.

10. The shipping containers must be marked with THIS END UP, and arrow labels, which indicate the proper upward position of the container, should be affixed to the cooler. A label containing the name and address of the shipper shall be placed on the outside of the container. Labels used in the shipment of hazardous materials (such as Cargo Only Air Craft, Flammable Solids, etc.) are not permitted to be on the outside of the container used to transport environmental samples and shall not be used.

4.4.3 Sample Custody

Sample collection and sample custody procedures are designed so that field custody of samples is maintained and documented. These procedures provide identification and documentation of the sampling event and the sample Chain-of-Custody from shipment of sample containers, through sample collection, to receipt of the sample by the subcontracted laboratory. When used in conjunction with the laboratory's custody procedures and the sample container documentation, these data establish full legal custody and allow complete tracking of a sample from preparation and receipt of sample container to sample collection, preservation, and shipping through laboratory receipt, sample analysis, and data validation. The chain-of-custody is defined as the sequence of persons who have the item in custody.

Field custody procedures are described below. Sample collection procedures concerning sample identification and documentation, field log book, sample containers, sample packing, and sample shipping are described.

The persons responsible for sample custody, and a brief description of their duties, are as follows:

- Laboratory Sample Custodian or Commercial Supplier: Verifies that the bottleware is certified clean; arranges for bottleware shipment to field sampling personnel or the contractor's equipment shop.
- Field Staff: Receives sample bottleware from laboratory, inspects bottleware for physical integrity; retains shipping invoice or packing list from shipping courier as documentation of transfer of bottleware; collects and preserves samples; retains bottleware and samples under custody until sample shipment; relinquishes samples to shipping courier or to lab representative.
- Laboratory Project Manager: Verifies reported laboratory analyses to the sample Chain-of-Custody Record; assures that chain-of-custody documentation is incorporated into the project file.

A sample or other physical evidence is in custody if it is:

- in the field investigator's, transferee's, or lab technician's actual possession; or
- in the field investigator's, transferee's, or lab technician's view, after being in his/her physical possession; or
- in the field investigator's, transferee's, or lab technician's physical possession and then he/she secured it to prevent tampering; or
- placed in a designated secure area.

The field Chain-of-Custody Record is used to record the custody of all samples or other physical evidence collected and maintained. This form shall not be used to document the collection of split or duplicate samples. The Chain-of-Custody Record also serves as a sample logging mechanism for the analytical laboratories' sample custodian.

The following information must be supplied in the indicated spaces in detail to complete the field Chain-of-Custody Record:

- project-specific information, including the project number and project name;
- signatures of all samplers and/or the sampling team leader in the designated signature block;
- sampling station number, date, and time of sample collection, grab or composite sample designation, and sample preservation type included on each line (each line shall contain only those samples collected at a specific location);

- sampling team leader's name recorded in the right or left margin of the Chain-of-Custody Record when samples collected by more than one sampling team are included on the same form;
- total number of sample containers listed in the indicated space for each sample and the total number of individual containers for each type of analysis under the indicated media or miscellaneous columns (note that it is impossible to have more than one media type per sample);
- documentation of the transfer of samples listed on the Chain-of-Custody Record by the field investigator and subsequent transferee(s) in the spaces provided at the bottom of the form (both the person relinquishing the samples and the person receiving them must sign the form; provide the date and time that this occurred in the proper space on the form; and usually, the last person receiving the samples or evidence should be a laboratory sample custodian); and
- air bill numbers or registered or certified mail serial numbers recorded in the remarks column at the bottom of the form.

The Chain-of-Custody Record is a serialized document. Once the Chain-of-Custody Record is completed, it becomes an accountable document and must be maintained in the project file. The suitability of any other form for chain-of-custody should be evaluated upon its inclusion of all of the above information in a legible format.

5.0 REPORTING AND SCHEDULE

Field work is tentatively scheduled for June, but is largely dependent on water levels in the LVR and weather conditions. The laboratory toxicity tests are 10 days (*C. dilutus*) and 28 days (*H. azteca*) in duration. Once the toxicity reports are received from the laboratory, Geosyntec will summarize the results and prepare a report to be included as an attachment to the BERA. The toxicity test results will be integrated into the BERA as an additional line of evidence to evaluate the overall status of the ecological community of the LVR. The RI Report will subsequently be revised accordingly.

6.0 REFERENCES

Besser, JM, WG Brumbaugh CD Ivey, CG Ingersoll, and PW Moran. 2008. Biological and Chemical Characterization of Metal Bioavailability in Sediments from Lake Roosevelt, Columbia River, Washington, USA. Arch Environ Contam Toxicol. 54:557–570.

Clark, DG, MR Palermo, and TC Sturgis. 2001. Subaqueous Cap Design: Selection of Bioturbation Profiles, Depths, and Rates. DOER Technical Notes Collection. EDRC TN-DOER-C21. Unite States Army Engineers Research and Development Center, Vicksburg, MS.

Geosyntec. 2007. Final Remedial Investigation/Feasibility Study Planning Documents, Work Plan (Operable Unit 1). Matthiessen and Hegeler Zinc Company Site, LaSalle, Illinois. July.

Geosyntec. 2010. Draft Remedial Investigation Report. Matthiessen and Hegeler Zinc Company Site, LaSalle, Illinois. May.

Suter, GW II. 1996. Risk Characterization for Ecological Risk Assessment of Contaminated Sites. Oak Ridge National Laboratory. Oak Ridge, TN. ES/ER/TM-200.

Suter, GW II and CL Tsao. 1996. Toxicological Benchmarks for Screening Potential Contaminants of Concern for Effects on Aquatic Biota: 1996 Revision. Oak Ridge National Laboratory, Oak Ridge, TN. ES/ER/TM-96/R2.

USEPA (United States Environmental Protection Agency). 2000. Methods for Measuring the Toxicity and Bioaccumulation of Sediment-associated Contaminants in Freshwater Invertebrates. EPA 600/R-99/064. March.